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IS : 9316 (Part 8) - 1987

(Reaffirmed 2003)

Indian Standard

METHODS OF TEST FOR RUBBER LATEX

PART 8 DETERMINATION OF TOTAL IRON

[RL : 8]

UDC 678·4·031 : 543 : 546·72

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *November* 1987

Indian Standard

METHODS OF TEST FOR RUBBER LATEX

PART 8 DETERMINATION OF TOTAL IRON

[RL : 8]

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Indian Standard

METHODS OF TEST FOR RUBBER LATEX

PART 8 DETERMINATION OF TOTAL IRON

[RL : 8]

0. FOREWORD

0.1 This Indian Standard (Part 8) was adopted by the Indian Standards Institution on 27 February 1987, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Test methods for rubber latex had been originally covered in the following Indian Standards:

For natural rubber latex

IS : 3708 (Part 1)-1966*

IS : 3708 (Part 2)-1968†

For styrene butadiene rubber latex

IS : 4511 (Part 1)-1967‡

Since some of the test methods covered in above standards were common, the concerned Committee had decided some years ago to unify and publish a separate series of methods of test which would be applicable to all types of latices — natural as well as synthetic. Accordingly, the following six test methods had been covered under IS : 9316:

IS : 9316 Methods of test for rubbe latex:

Part 1-1979 Determination of surface tension

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*Methods of test for natural rubber latex: Part 1 Dry rubber content, sludge content, density, total alkalinity, KOH-number, mechanical stability, volatile fatty acid number, *p*H, total nitrogen, total copper, total iron, total manganese and total ash.

†Methods of test for natural rubber latex, Part 2.

‡Methods of tests for styrene-butadiene rubber (SBR) latices: Part 1 Determination of dry polymer, *p*H density residual styrene, bound styrene and soap content.

Part 2-1979 Determination of viscosity
Part 3-1979 Determination of coagulum content
Part 4-1979 Determination of total solids content
Part 5-1979 Drawing of samples
Part 6-1982 Determination of *p*H

0.2.1 As a result of further rethinking on the subject, it has now been decided to re-designate the test methods common to natural and synthetic rubber latices as RL series; test methods for natural rubber latex as NRL series and test methods for styrene-butadiene rubber latex as SBRL series. Consequently, test methods for rubber latex have been rationalized into the following three series:

a) IS : 9316 Unified methods of test applicable to both natural and synthetic rubber latices — RL series;

b) IS : 3708 Methods of test applicable to natural rubber latex — NRL series; and

c) IS : 4511 Method of test applicable to styrene-butadiene rubber latex — SBRL series.

0.3 The existing Indian Standards under IS : 3708 (Parts 1* and 2†), IS : 4511 (Part 1‡) and IS : 9316 (Parts 1 to 6) are being gradually replaced by separate standards under the above three series, designated by the appropriate NRL, SBRL, or RL series, respectively.

0.3.1 This method originally covered under NRL : 14 of IS : 3708 (Part 1)-1966, is now being covered in this standard.

0.4 In order to facilitate cross-reference, it has been decided to retain the original discrete NRL, SBRL and RL series numbers assigned to various test methods; in IS : 3708 (Parts 1* and 2†), IS : 4511 (Part 1‡) and IS : 9316 (Parts 1 to 6) in the revised parts of IS : 3708, IS : 4511 and IS : 9316 respectively.

0.4.1 For proper referencing of the existing test methods and the new methods under revision, a statement showing corresponding methods is given in Appendix A.

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*Methods of test for natural rubber latex: Part 1 Dry rubber content, sludge content, density, total alkalinity, KOH-number, mechanical stability, volatile fatty, *p*H, total nitrogen, total copper, total iron, total manganese and total ash.

†Methods of test for natural rubber latex, Part 2.

‡Methods of tests for styrene-butadiene rubber (SBR) latices: Part 1 Determination of dry polymer, *p*H, density, residual styrene, bound styrene and soap content.

0.5 In preparing the above series, the need to align the test methods with the corresponding ISO Standards/DIS/DP wherever available has also been taken into account for updating the test methods. In the preparation of this standard, assistance has been derived from ISO 1657-1975 'Raw rubber and rubber latex — Determination of iron content — 1, 10-phenanthroline photometric method', issued by International Organization for Standardization (ISO).

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 8) prescribes a method for the determination of total iron content in uncompounded natural rubber latex and uncompounded synthetic rubber latex which do not contain chlorine.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

3. OUTLINE OF THE METHOD

3.1 Dried latex solids are ashed in a crucible. The ash is extracted with hydrochloric acid and the solution is made up to standard volume. After adjustment of the *p*H by the addition of buffer solution, an aliquot part of the solution is treated with hydroxylammonium chloride and 1, — 10-phenanthroline with which the iron forms a coloured complex. The absorbance of this solution is measured photometrically, which is proportional to the concentration of iron.

4. APPARATUS

4.1 Electrophotometer or Spectrophotometer — capable of measuring absorbance at approximately 510 nm.

4.2 Silica or Porcelain Crucible — nominal capacity 50 to 80 ml.

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*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

4.3 Muffle Furnace — capable of maintaining a temperature of 525 ± 25°C.

4.4 Asbestos Board — approximately 100 mm square and 6 mm thick, with a hole in the centre to support the crucible so that approximately two-thirds projects below the board.

4.5 Volumetric Flasks — 50, 100, 1 000 ml.

5. REAGENTS

5.1 Hydrochloric Acid — concentrated (*see* IS : 265-1976*).

5.2 1, 10-Phenanthroline Solution — Dissolve 0·5 g of 1, 10-phenanthroline monohydrate in hot water and dilute the olution to 500 ml.

5.3 Hydroxylammonium Chloride Solution — Dissolve 10 g of hydroxylammonium chloride in 100 ml of water.

5.4 Buffer Solution — Dissolve 164 g of anhydrous sodium acetate in approximately 250 ml of water and to the solution add 28·5 ml of glacial acetic acid (sp gr 1·05). Dilute this mixture to 500 ml and filter immediately before use if it is cloudy. If this buffer solution gives highly coloured reference solutions in the preparation of the calibration curve, an alternative buffer solution may be prepared by dissolving 80 g of sodium hydroxide or 106 g of anhydrous sodium carbonate in 200 ml of water, adding 142·5 ml of glacial acetic acid, (sp gr 1·05) and diluting the solution to 500 ml.

5.5 Standard Iron Solution (1 ml Contains 0·1 mg of Fe) — Dissolve 0·702 1 g of ferrous ammonium sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$] in water in a 1 000-ml volumetric flask. Add 3 ml of concentrated hydrochloric acid (sp gr 1·19) and dilute to the mark with water. This solution should remain stable for at least one month.

5.6 Standard Iron Working Solution (1 ml Contains 0·01 mg of Fe) — Pipette 10 ml of the standard iron solution (**5.5**) into a 100-ml volumetric flask and dilute to the mark with water. This solution shall be freshly prepared from the stock solution (**5.5**) when required.

6. PROCEDURE

6.1 Preparation of Test Portion — For the determination of iron in latex, take a portion of thoroughly mixed latex containing at least 10 g

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*Specification for hydrochloric acid (*second revision*).

of total solids and dry to constant mass [*see* IS : 9316 (Part 4)-1979*]. Homogenize the rubber so obtained by passing it a few times between the cold rolls of a laboratory mill to produce a thin sheet.

NOTE — At all stages of sample preparation, care shall be taken to avoid contamination with iron, particularly from rusty knives, scissors, mills, and other equipment.

6.2 Preparation of Calibration Curve — Make up a series of standard matching solutions in 50-ml volumetric flasks, each containing 1 ml of concentrated hydrochloric acid, 10 ml of buffer solution (**5.4**) 1 ml of hydroxylammonium chloride solution (**5.3**) and 10 ml of 1, 10-phenanthroline solution (**5.2**). To these solutions, add portions of the standard iron working solution (**5.6**), ranging from 0 to 20 ml (for example 0; 0·5; 5; 10; 15; 20 ml) and dilute the solutions with water to the mark. Allow to stand for 15 min, then decant each solution into the cell of the electrophotometer or spectrophotometer and measure its absorbance at the absorption maximum (about 510 nm). Correct the reading by subtracting the absorbance of the solution containing no added iron. If the absorbance is measured on a double-beam instrument, place the cell containing the blank solution in the reference beam and measure the absorbance of each standard matching solution against that of the solution containing no added iron. Plot the reading thus obtained for each solution against the appropriate concentration of iron to give the calibration curve, which shall be checked periodically according to local conditions and the type of instrument used.

6.3 Determination

6.3.1 Cut a 10 g test portion of the dried latex into small pieces. Transfer to the unetched crucible and weigh to the nearest 0·01 g. Support the crucible in the hole cut in the asbestos board. Heat gently with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to the muffle furnace maintained at a temperature of 525 ± 25°C.

6.3.2 Alternatively, wrap the weighed sample in a piece of ashless filter paper about 150 mm in diameter and transfer to the crucible. Place the crucible and its contents into the furnace at 525 ± 25°C and close the furnace door. The furnace door shall not be opened during the first hour because of the risk of igniting inflammable gases. When all the carbon has been oxidized remove the crucible and allow to cool.

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*Methods of test for rubber latex: Part 4 Determination of total solids content.

6.3.3 In parallel with the determination and following the same procedure, carry out a blank determination using similar filter paper and crucible and using the same quantities of all the reagents as used for the determination.

6.3.4 Add 5 ml of hydrochloric acid and 5 ml of water to the crucible and digest the mixture on a steam bath for 30 to 60 min. If the solution has a deep yellow colour, indicating the presence of much iron, add a further 5 ml of the hydrochloric acid and digest for a further 30 min. Filter the solution through a sintered glass crucible (porosity grade 2 or 3). Collect the filtrate in a 50-ml volumetric flask and dilute to the mark.

6.3.5 Transfer an aliquot part of this solution containing not more than 2 ml of hydrochloric acid to a 50-ml volumetric flask. Add 10 ml of the buffer solution, then 1 ml of hydroxylammonium chloride solution and 10 ml of 1, 10-phenanthroline solution. Dilute to the mark with water and allow to stand at room temperature for 15 min.

6.3.6 Measure the absorbance of the solution at the wavelength used for preparing the calibration curve. Correct the reading for the test solution by subtracting the value for the absorbance of the blank solutions. If the absorbance is measured on a double-beam instrument, place the cell containing the blank solution in the reference beam and measure the absorbance of the test solution against the blank solution.

NOTE — All precautions and safeguards required for the carrying out of trace metal analysis shall be observed.

7. EXPRESSION OF RESULTS

7.1 By means of the calibration curve, determine the concentration of iron corresponding to the corrected reading, and from this calculate the iron content of the test portion. Express the result as parts per million (ppm) of iron (Fe) calculated by mass.

APPENDIX A

(*Clause* 0.4.1)

TABLE SHOWING CORRESPONDENCE OF THE VARIOUS METHODS OF TEST COVERED IN THE EXISTING IS : 9316 (PARTS 1 TO 5)-1979, IS : 9316 (PART 6)-1982, IS : 3708 (PART 1)-1966, IS : 3708 (PART 2)-1968, IS : 4511 (PART 1)-1967 *WITH* THE REVISION/PROPOSED REVISION OF IS : 9316, IS : 3708 AND IS : 4511

Existing Test Methods			Proposed Revision		Remarks
Test Method	IS No.	Part (Series)	IS No.	Part (Series)	
(1)	(2)	(3)	(4)	(5)	(6)
RL Series					
Determination of surface tension	IS : 9316-1979	Part 1	IS : 9316-1987	Part 1 (RL : 1)	
Determination of viscosity	IS : 9316-1979	Part 2	IS : 9316-1987	Part 2 (RL : 2)	
Determination of coagulum content	IS : 9316-1979	Part 3	IS : 9316-1987	Part 3 (RL : 3)	
Determination of total solids content	IS : 9316-1979	Part 4	IS : 9316	Part 4 (RL : 4)	Under revision
Drawing of samples	IS : 9316-1979	Part 5	IS : 9316	Part 5 (RL : 5)	
Determination of *p*H	IS : 9316-1982	Part 6	IS : 9316	Part 6 (RL : 6)	
Determination of total copper	IS : 3708-1966	Part 1 (NRL : 13)	IS : 9316-1987	Part 7 (RL : 7)	
Determination of total iron	IS : 3708-1966	Part 1 (NRL : 14)	IS : 9316-1987	Part 8 (RL : 8)	
Determination of total manganese	IS : 3708-1966	Part 1 (NRL : 15)	IS : 9316-1987	Part 9 (RL : 9)	
NRL Series					
Determination of dry rubber content	IS : 3708-1966	Part 1 (NRL : 1)	IS : 3708-1985	Part 1 (NRL : 1)	
Determination of sludge content	IS : 3708-1966	Part 1 (NRL : 5)	IS : 3708-1985	Part 2 (NRL : 5)	
Determination of density	IS : 3708-1966	Part 1 (NRL : 6)	IS : 3708-1985	Part 3 (NRL : 6)	
Determination of total alkalinity	IS : 3708-1966	Part 1 (NRL : 7)	IS : 3708-1985	Part 4 (NRL : 7)	

(*Continued*)

EXISTING TEST METHODS			PROPOSED REVISION		REMARKS
Test Method	IS No.	Part (Series)	IS No.	Part (Series)	
(1)	(2)	(3)	(4)	(5)	(6)
Determination of KOH-number	IS : 3708-1966	Part 1 (NRL : 8)	IS : 3708-1985	Part 5 (NRL : 8)	
Determination of mechanical stability	IS : 3708-1966	Part 1 (NRL : 9)	IS : 3708-1985	Part 6 (NRL : 9)	
Determination of volatile fatty acid number	IS : 3708-1966	Part 1 (NRL : 10)	IS : 3708-1986	Part 7 (NRL : 10)	
Determination of total nitrogen	IS : 3708-1966	Part 1 (NRL : 12)	IS : 3708	Part 8 (NRL : 12)	
Determination of total ash	IS : 3708-1966	Part 1 (NRL : 16)	IS : 3708-1986	Part 9 (NRL : 16)	
Determination of boric acid	IS : 3708-1968	Part 2 (NRL : 17)	IS : 3708-1986	Part 10 (NRL : 17)	
Determination of magnesium	IS : 3708-1968	Part 2 (NRL : 18)	IS : 3708-1986	Part 11 (NRL : 18)	
SBRL Series					
Determination of dry polymer	IS : 4511-1967	Part 1 (SBRL : 1)	IS : 4511-1986	Part 1 (SBRL : 1)	
Determination of density	IS : 4511-1967	Part 1 (SBRL : 6)	IS : 4511-1986	Part 2 (SBRL : 6)	
Determination of volatile unsaturates	IS : 4511-1967	Part 1 (SBRL : 8)	IS : 4511	Part 3 (SBRL : 8)	
Determination of bound styrene	IS : 4511-1967	Part 1 (SBRL : 9)	IS : 4511	Part 4 (SBRL : 9)	
Determination of soap content	IS : 4511-1967	Part 1 (SBRL : 10)	IS : 4511	Part 5 (SBRL : 10)	
Determination of high-speed mechanical stability	—	—	IS : 4511	Part 6 (SBRL : 11)	

(*Continued from page* 2)

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INTERNATIONAL SYSTEM OF UNITS (SINUITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane Angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2